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(54) Title: STEAM EXPLOSION TREATMENT WITH ADDITION OF CHEMICALS (57) Abstract De-inked recycled fibers modified by steam are able to form handsheets with higher bulk, lower tensile and a higher porosity. Consequently, the modified fibers show significant improvement in softness which is fundamental importance in tissue manufacturing. Bleach can be added to compensate for the brightness reduction caused by the steam explosion treatment.		

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STEAM EXPLOSION TREATMENT WITH ADDITION OF CHEMICALS

Field of the Invention

The invention is directed generally to the recycling of paper fibers and is directed more specifically to the steam explosion recycling of previously de-inked fibers. The invention is further directed to tissues manufactured from said recycled fibers.

5

Background of the Invention

The use of steam or explosive decompression to disintegrate or fiberize wood fibers is well known in the art. For example, the Mason patents dating from the 1920s and 1930s disclose the general techniques of steam explosion treatments. These patents include U. S. Patent Nos. 1,586,159, 1,578,609, 1,655,618, 1,824,221, 1,872,996 and 1,922,313. All of
10 said references are directed generally to the disintegration of cellulosic materials.

Later patents are directed to incremental improvements and refinements in steam explosion treatments. For example, U. S. Patent No. 2,516,847 to Boehm is directed to a means of sizing the exploded fibers. Mitscherling, U. S. Patent No. 1,793,711, adds the
15 wrinkle of using a vacuum source to remove volatile resins prior to the pressurization and explosive decompression steps. Birdseye, U. S. Patent No. 2,711,369, refines the explosive decompression treatment by adding a series of explosive steps. Apparently this serves to more evenly disintegrate the fibers. This series of explosive steps permits the use of lower pressures and temperatures.

20 Mamers, et al., U. S. Patent No. 4,163,687, is directed to a uniquely designed nozzle for assisting the liberation of fibers from cellulosic material during explosive defibration. The nozzle has a plurality of internal bars which provide a tortuous path for material passing through. O'Connor, U. S. Patent No. 3,707,436, adds the wrinkle of using ammonia instead of steam. Apparently compounds such as ammonia are effective at
25 swelling and platisizing wood. Morgan, U. S. Patent No. 2,234,188, is directed to production of light-colored cellulosic fiber. This is accomplished by first treating the chips or other small pieces of wood with an alkaline sulphite or alkaline metal such as sodium sulphite or potassium sulphite.

30 However, steam explosion processes often have accompanying problems. For example, the major problems accompanying previous processes using explosive

decompression are believed to have been the degradation due to the oxidation of wood and acid hydrolysis leading to loss in brightness, deterioration of fiber and paper properties and loss of yield. The approach adopted by this invention is therefore to attempt to curtail hydrolytic and oxidative wood degradation and thereby to protect against loss of yield, brightness and fiber strength. The loss of fiber strength will be particularly great if the degree of polymerization of the cellulose falls below the critical value which is about 500-600. Hydrolytic degradation will also cause yield loss due mainly to degradation of hemi-cellulose.

Kokta, U.S. Patent No. 4,798,651 addresses some of these concerns by disclosing a process for producing high-yield pulp and for recovering waste paper. Waste paper is recycled by breaking down the ink particles during the steam explosion process. However, nothing in the past indicates that the steam process can be used to modify pulps for tissue manufacture.

Summary of the Invention

The invention is directed to the preparation of a modified version of de-inked recycled fibers. This modification comprises steam explosion of the recycle fibers. These modified fibers are able to form handsheets with higher bulk, lower tensile and a higher porosity. Consequently, the modified fibers show significant improvement in softness which is fundamental importance in tissue manufacturing. Bleach can be added to compensate for the brightness reduction caused by the steam explosion treatment.

Accordingly, the invention is found in a process for recycling waste paper by subjecting de-inked fibers to steam explosion treatment; said process optionally including a concurrent bleaching step to improve brightness.

Another embodiment of the invention concerns recycled waste paper created by subjecting de-inked fibers to steam explosion treatment and optionally including a concurrent bleaching step to improve brightness.

A further embodiment is found in a process for manufacturing tissues which exhibit increased bulk, increased porosity and decreased tensile strength.

Another embodiment is found in a tissue which exhibits increased bulk, increased porosity and decreased tensile strength.

Detailed Description of the Invention

It has been discovered that, by using a steam explosion process for treating cellulosic fibers, and by using appropriate treatment conditions, modified cellulosic fibers exhibiting desired properties may be prepared by an efficient and effective process.

5 A wide variety of cellulosic fibers can be employed in the process of the present invention. Illustrative cellulosic fibers include, but are not limited to, wood and wood products, such as wood pulp fibers; non-woody paper-making fibers from cotton, from straws and grasses, such as rice and esparto, from canes and reeds, such as bagasse, from bamboos, from stalks with bast fibers, such as jute, flax, kenaf, cannabis, linen and ramie,
10 and from leaf fibers, such as abaca and sisal. It is also possible to use mixtures of one or more cellulosic fibers. Suitably, the cellulosic fiber used is from a wood source. Suitable wood sources include softwood sources such as pines, spruces, and firs, and hardwood sources such as oaks, eucalyptuses, poplars, beeches, and aspens.

As used herein, the term "fiber" or "fibrous" is meant to refer to a particulate material
15 wherein the length to diameter ratio of such particulate material is greater than about 10. Conversely, a "nonfiber" or "nonfibrous" material is meant to refer to a particulate material wherein the length to diameter ratio of such particulate material is about 10 or less.

It is generally desired that the cellulosic fibers used herein be wettable. As used herein, the term "wetable" is meant to refer to a fiber or material which exhibits a water in
20 air contact angle of less than 90°. Suitably, the cellulosic fibers useful in the present invention exhibit a water in air contact angle between about 10° to about 50° and more suitably between about 20° to about 30°. Suitably, a wettable fiber refers to a fiber which exhibits a water in air contact angle of less than 90°, at a temperature between about 0°C and about 100°C, and suitably at ambient conditions, such as about 23°C.

25 Suitably cellulosic fibers are those which are naturally wettable. However, naturally nonwettable fibers can also be used. It is possible to treat the fiber surfaces by an appropriate method to render them more or less wettable. When surface treated fibers are employed, the surface treatment is desirably nonfugitive; that is, the surface treatment desirably does not wash off the surface of the fiber with the first liquid insult or contact.
30 For the purposes of this application, a surface treatment on a generally nonwettable fiber will be considered to be nonfugitive when a majority of the fibers demonstrate a water in air contact angle of less than 90° for three consecutive contact angle measurements, with drying between each measurement. That is, the same fiber is subjected to three separate

contact angle determinations and, if all three of the contact angle determinations indicate a contact angle of water in air of less than 90°, the surface treatment on the fiber will be considered to be nonfugitive. If the surface treatment is fugitive, the surface treatment will tend to wash off of the fiber during the first contact angle measurement, thus exposing the nonwettable surface of the underlying fiber, and will demonstrate subsequent contact angle measurements greater than 90°. Beneficial wettability agents include polyalkylene glycols, such as polyethylene glycols. The wettability agent is used in an amount comprising beneficially less than about 5 weight percent, suitably less than about 3 weight percent, and more suitably less than about 2 weight percent, of the total weight of the fiber, material, or absorbent structure being treated.

In the present invention, it is desired that the cellulosic fibers be used in a form wherein the cellulosic fibers have already been refined into a pulp. As such, the cellulosic fibers will be substantially in the form of individual cellulosic fibers although such individual cellulosic fibers may be in an aggregate form such as a pulp sheet. The current process, then, is in contrast to known steam explosion processes that generally treat cellulosic fibers that are typically in the form of virgin wood chips or the like. Thus, the current process is a post-pulping, cellulosic fiber modifying process as compared to known steam explosion processes that are generally used for high-yield pulp manufacturing or waste-recycle processes.

The cellulosic fibers used in the steam explosion process are desirably low yield cellulosic fibers. As used herein, "low yield" cellulosic fibers are those cellulosic fibers produced by pulping processes beneficially providing a yield of about 85 percent or less, suitably of about 80 percent or less, and more suitably of about 55 percent or less. In contrast, "high yield" cellulosic fibers are those cellulosic fibers produced by pulping processes beneficially providing a yield of about 85 percent or greater. Such pulping processes generally leave the resulting cellulosic fibers with high levels of lignin.

In the process of the present invention, it has been discovered that the use of steam explosion alone can be sufficient to effectively modify cellulosic fibers such that the modified cellulosic fibers exhibit desired properties, particularly desired liquid absorbency properties. In general, it is desired that the cellulosic fibers are cooked in a saturated steam environment that is substantially free of air. The presence of air in the pressurized cooking environment may result in the oxidation of the cellulosic fibers. As such, it is desired that the cellulosic fibers are cooked in a saturated steam environment that beneficially

comprises less than about 5 weight percent, suitably less than about 3 weight percent, and more suitably less than about 1 weight percent of air, based on the total weight of the gaseous environment present in the pressurized cooking environment.

5 The individual cellulosic fibers are steam cooked at a high temperature and at a high pressure. In general, any combination of high pressure, high temperature, and time which is effective in achieving a desired degree of modification, without undesirable damage to the cellulosic fibers, so that the cellulosic fibers exhibit the desired liquid absorbency properties as described herein, is suitable for use in the present invention.

10 Generally, if the temperature used is too low, there will not be a substantial and/or effective amount of modification of the cellulosic fibers that occurs. Also, generally, if the temperature used is too high, a substantial degradation of the cellulosic fibers may occur which will negatively affect the properties exhibited by the treated cellulosic fibers. As such, as a general rule, the cellulosic fibers will be treated at a temperature within the range beneficially from about 130°C to about 250°C, suitably from about 150°C to about 225°C, 15 more suitably from about 160°C to about 225°C, and most suitably from about 160°C to about 200°C.

20 Generally, the cellulosic fibers will be subjected to an elevated superatmospheric pressure over a time period within the range of from about 0.1 minute to about 30 minutes, beneficially from about 0.5 minute to about 20 minutes, and suitably from about 1 minute to about 10 minutes. In general, the higher the temperature employed, the shorter the period of time generally necessary to achieve a desired degree of modification of the cellulosic fibers. As such, it may be possible to achieve essentially equivalent amounts of modification for different cellulosic fiber samples by using different combinations of high temperatures and times.

25 Generally, if the pressure used is too low, there will not be a substantial and/or effective amount of modification of the cellulosic fibers that occurs. Also, generally, if the pressure used is too high, a substantial degradation of the cellulosic fibers may occur which will negatively affect the properties exhibited by the crosslinked cellulosic fibers. As such, as a general rule, the cellulosic fibers will be treated at a pressure that is superatmospheric (i.e. above normal atmospheric pressure), beneficially within the range from about 40 to 30 about 405 pounds per square inch, suitably from about 40 to about 230 pounds per square inch, and more suitably from about 90 to about 230 pounds per square inch.

As used herein, "consistency" is meant to refer to the concentration of the cellulosic fibers present in an aqueous mixture. As such, the consistency will be presented as a weight percent representing the weight amount of the cellulosic fibers present in an aqueous mixture divided by the total weight amount of cellulosic fibers and water present in such mixture, multiplied by 100.

In general, the cellulosic fibers may be used in the process of the present invention in either a dry or a wet state. However, it may be desirable to prepare an aqueous mixture comprising the cellulosic fibers wherein the aqueous mixture is agitated, stirred, or blended to effectively disperse the cellulosic fibers throughout the water. In one embodiment of the present invention, it is desired that the cellulosic fibers be steam cooked when the cellulosic fibers are in the form of aqueous pulp mixture that beneficially has a consistency of between about 10 to about 100 weight percent, suitably between about 20 to about 80 weight percent, and more suitably between about 25 to about 75 weight percent cellulosic fibers, based on the total weight percent of the aqueous pulp mixture.

The cellulosic fibers are typically mixed with an aqueous solution beneficially comprising at least about 30 weight percent water, suitably about 50 weight percent water, more suitably about 75 weight percent water, and most suitably 100 weight percent water. When another liquid is employed with the water, such other suitable liquids include methanol, ethanol, isopropanol, and acetone. However, the use or presence of such other non-aqueous liquids may impede the formation of an essentially homogeneous mixture such that the cellulosic fibers do not effectively disperse into the aqueous solution and effectively or uniformly mix with the water. Such a mixture should generally be prepared under conditions that are sufficient for the cellulosic fibers and water to be effectively mixed together. Generally, such conditions will include using a temperature that is between about 10°C to about 100°C.

In general, cellulosic fibers are prepared by pulping or other preparation processes in which the cellulosic fibers are present in an aqueous solution. For use in the steam explosion treatment of the present invention, therefore, it may be possible to use an aqueous solution directly from such preparation processes without having to separately recover the cellulosic fibers.

After steam cooking the cellulosic fibers, the pressure is released and the cellulosic fibers are exploded into a release vessel. The equipment or method used to treat the cellulosic fibers with steam explosion is generally not critical. Suitable equipment and

methods for steam explosion may be found, for example, in Canadian Patent No. 1,070,537, dated Jan. 29, 1980; Canadian Patent No. 1,070,646, dated Jan. 29, 1980; Canadian Patent No. 1,119,033, dated Mar. 2, 1982; Canadian Patent No. 1,138,708, dated Jan. 4, 1983; and US Patent 5,262,003, issued November 16, 1993, all of which are
5 incorporated herein in their entirety by reference.

The steam explosion process generally causes the cellulosic fibers to become modified. Without intending to be bound hereby, it is believed that the steam explosion process causes the cellulosic fibers to undergo a curling phenomenon. The steam exploded cellulosic fibers, in addition to being modified, have been discovered to exhibit improved
10 properties that make such steam exploded cellulosic fibers suitable for use in liquid absorption or liquid handling applications.

Cellulosic fibers suitable for use in the present invention are generally without a substantial amount of curl prior to the steam explosion process. After such steam explosion process, the treated cellulosic fibers will generally exhibit a desired level of stable curl. As
15 such, the process of the present invention generally does not require the use of any additional additives to the cellulosic fibers during the steam explosion process or any post-treatment steps after the steam explosion of the fibers to achieve the desired curls.

In one embodiment of the present invention, the cellulosic fibers will be considered to be effectively treated by the steam explosion process when the cellulosic fibers exhibit
20 an effective Wet Curl value. The curl of a fiber may be quantified by a curl value which measures the fractional shortening of a fiber due to kink, twists, and/or bends in the fiber. For the purposes of this invention, a fiber's curl value is measured in terms of a two dimensional plane, determined by viewing the fiber in a two dimensional plane. To determine the curl value of a fiber, the projected length of a fiber as the longest dimension
25 of a two dimensional rectangle encompassing the fiber, l , and the actual length of the fiber, L , are both measured. An image analysis method may be used to measure L and l . A suitable image analysis method is described in U.S. Patent 4,898,642, incorporated herein in its entirety by reference. The curl value of a fiber can then be calculated from the following equation:

30
$$\text{Curl Value} = (L/l) - 1$$

Depending on the nature of the curl of a cellulosic fiber, such curl may be stable when the cellulosic fiber is dry but may be unstable when the cellulosic fiber is wet. The cellulosic fibers prepared according to the process of the present invention have been found

to exhibit a substantially stable fiber curl when wet. This property of the cellulosic fibers may be quantified by a Wet Curl value, as measured according to the test method described herein, which is a length weighted mean curl average of a designated number of fibers, such as about 4000, from a fiber sample. As such, the Wet Curl value is the summation of the individual wet curl values for each fiber multiplied by the fiber's actual length, L, divided by the summation of the actual lengths of the fibers. It is hereby noted that the Wet Curl value, as determined herein, is calculated by only using the necessary values for those fibers with a length of greater than about 0.4 millimeter.

As used herein, the cellulosic fibers will be considered to be effectively treated by the steam explosion treatment when the cellulosic fibers exhibit a Wet Curl value that is greater than about 0.2, beneficially between about 0.2 to about 0.4, more beneficially between about 0.2 to about 0.35, suitably between about 0.22 to about 0.33, and suitably between about 0.25 to about 0.33. In contrast, cellulosic fibers that have not been treated generally exhibit a Wet Curl value that is less than about 0.2.

After the cellulosic fibers have been effectively steam exploded, the treated cellulosic fibers are suitable for use in a wide variety of applications. However, depending on the use intended for the treated cellulosic fibers, such treated cellulosic fibers may be washed with water. If any additional processing procedures are planned because of the specific use for which the treated cellulosic fibers are intended, other recovery and post-treatment steps are also well known.

The cellulosic fibers treated according to the process of the present invention are suited for use in disposable absorbent products such as diapers, adult incontinent products, and bed pads; in catamenial devices such as sanitary napkins, and tampons; other absorbent products such as wipes, bibs, wound dressings, and surgical capes or drapes; and tissue-based products such as facial or bathroom tissues, household towels, wipes and related products. Accordingly, in another aspect, the present invention relates to a disposable absorbent product comprising the cellulosic fibers treated according to the process of the present invention.

In one embodiment of the present invention, the treated fibers prepared according to the process of the present invention are formed into a handsheet which might represent a tissue-based product. Such a handsheet may be formed by either a wet-laid or an air-laid process. A wet-laid handsheet may be prepared according to the method disclosed in the Test Methods section herein.

It has been discovered that a wet-laid handsheet prepared from the treated cellulosic fibers prepared according to the process of the present invention may exhibit a density that is lower than a wet-laid handsheet prepared from cellulosic fibers that have not been treated according to the process of the present invention.

5 It has also been discovered that a wet-laid handsheet prepared from the treated cellulosic fibers prepared according to the process of the present invention may exhibit a liquid wicking time that is faster than a wet-laid handsheet prepared from cellulosic fibers that have not been treated according to the process of the present invention.

10 It has also been discovered that a wet-laid handsheet prepared from the treated cellulosic fibers prepared according to the process of the present invention may exhibit a liquid wicking flux that is higher than a wet-laid handsheet prepared from cellulosic fibers that have not been treated according to the process of the present invention.

15 It has also been discovered that a wet-laid handsheet prepared from the treated cellulosic fibers prepared according to the process of the present invention may exhibit an increased bulk and higher absorbent capacity than a wet-laid handsheet prepared from cellulosic fibers that have not been treated according to the process of the present invention.

In one embodiment of the present invention, the treated cellulosic fibers prepared according to the process of the present invention are formed into a fibrous matrix for
20 incorporation into an absorbent structure. A fibrous matrix may take the form of, for example, a batt of comminuted wood pulp fluff, a tissue layer, a hydroentangled pulp sheet, or a mechanically softened pulp sheet. An exemplary absorbent structure is generally described in copending US Patent application, Serial Number 60/008,994, which reference is incorporated herein in its entirety by reference.

25 A fibrous matrix useful in the present invention may be formed by an air-laying process or a wet-laid process, or by essentially any other process known to those skilled in the art for forming a fibrous matrix.

In one embodiment of the present invention, a disposable absorbent product is provided, which disposable absorbent product comprises a liquid-permeable topsheet, a
30 backsheet attached to the liquid-permeable topsheet, and an absorbent structure positioned between the liquid-permeable topsheet and the backsheet, wherein the absorbent structure comprises treated cellulosic fibers prepared using the process of the present invention.

Exemplary disposable absorbent products are generally described in US-A4,710,187; US-A-4,762,521; US-A-4,770,656; and US-A-4,798,603; which references are incorporated herein by reference.

Those skilled in the art will recognize materials suitable for use as the topsheet and
5 backsheet. Exemplary of materials suitable for use as the topsheet are liquid-permeable materials, such as spunbonded polypropylene or polyethylene having a basis weight of from about 15 to about 25 grams per square meter. Exemplary of materials suitable for use as the backsheet are liquid-impervious materials, such as polyolefin films, as well as vapor-
10 pervious materials, such as microporous polyolefin films.

Absorbent products and structures according to all aspects of the present invention
15 are generally subjected, during use, to multiple insults of a body liquid. Accordingly, the absorbent products and structures are desirably capable of absorbing multiple insults of body liquids in quantities to which the absorbent products and structures will be exposed during use. The insults are generally separated from one another by a period of time.

Test Procedures

Wet Curl

The Wet Curl value for fibers was determined by using an instrument which
20 rapidly, accurately, and automatically determines the quality of fibers, the instrument being available from OpTest Equipment Inc., Hawkesbury, Ontario, Canada, under the designation Fiber Quality Analyzer, OpTest Product Code DA93.

A sample of dried cellulosic fibers was obtained. The cellulosic fiber sample was
25 poured into a 600 milliliter plastic sample beaker to be used in the Fiber Quality Analyzer. The fiber sample in the beaker was diluted with tap water until the fiber concentration in the beaker was about 10 to about 25 fibers per second for evaluation by the Fiber Quality Analyzer.

An empty plastic sample beaker was filled with tap water and placed in the Fiber
30 Quality Analyzer test chamber. The <System Check> button of the Fiber Quality Analyzer was then pushed. If the plastic sample beaker filled with tap water was properly placed in the test chamber, the <OK> button of the Fiber Quality Analyzer was then pushed. The Fiber Quality Analyzer then performs a self-test. If a warning was not displayed on the screen after the self-test, the machine was ready to test the fiber sample.

The plastic sample beaker filled with tap water was removed from the test chamber and replaced with the fiber sample beaker. The <Measure> button of the Fiber Quality Analyzer was then pushed. The <New Measurement> button of the Fiber Quality Analyzer was then pushed. An identification of the fiber sample was then typed into the Fiber Quality Analyzer. The <OK> button of the Fiber Quality Analyzer was then pushed. The <Options> button of the Fiber Quality Analyzer was then pushed. The fiber count was set at 4,000. The parameters of scaling of a graph to be printed out may be set automatically or to desired values. The <Previous> button of the Fiber Quality Analyzer was then pushed. The <Start> button of the Fiber Quality Analyzer was then pushed. If the fiber sample beaker was properly placed in the test chamber, the <OK> button of the Fiber Quality Analyzer was then pushed. The Fiber Quality Analyzer then began testing and displayed the fibers passing through the flow cell. The Fiber Quality Analyzer also displayed the fiber frequency passing through the flow cell, which should be about 10 to about 25 fibers per second. If the fiber frequency is outside of this range, the <Stop> button of the Fiber Quality Analyzer should be pushed and the fiber sample should be diluted or have more fibers added to bring the fiber frequency within the desired range. If the fiber frequency is sufficient, the Fiber Quality Analyzer tests the fiber sample until it has reached a count of 4000 fibers at which time the Fiber Quality Analyzer automatically stops. The <Results> button of the Fiber Quality Analyzer was then pushed. The Fiber Quality Analyzer calculates the Wet Curt value of the fiber sample, which prints out by pushing the <Done> button of the Fiber Quality Analyzer.

Preparation of Wet-Laid Handsheet

A) Handsheet Forming:

A 7-1/2 inch by 7-1/2 inch handsheet has a basis weight of about 60 grams per square meter and was prepared using a Valley Handsheet mold, 8x8 inches. The sheet mold forming wire is a 90x90 mesh, stainless-steel wire cloth, with a wire diameter of 0.0055 inches. The backing wire is a 14"x14" mesh with a wire diameter of 0.021 inches, plain weave bronze. Taking a sufficient quantity of the thoroughly mixed stock to produce a handsheet of about 60 grams per square meter. Clamp the stock container of the sheet mold in position on the wire and allow several inches of water to rise above the wire. Add the measured stock and then fill the mold with water up to a mark of 6 inches above the

wire. Insert the perforated mixing plate into the mixture in the mold and slowly move it down and up 7 times. Immediately open the water leg drain valve. When the water and stock mixture drains down to and disappears from the wire, close the drain valve. Raise the cover of the sheet mold. Carefully place a clean, dry blotter on the formed fibers. Place the
5 dry couch roll at the front edge of the blotter. The fibers adhering to the blotter, are couched off the wire by one passage of the couching roll, without pressure, from front to back of wire.

B) Handsheet Pressing:

10 Place the blotter with the fiber mat adhering to it in the hydraulic press, handsheet up, on top of tow used, re-dried blotters. Two new blotters are placed on top of the handsheet. Close the press, clamp it and apply pressure to give a gauge reading that will produce 75 PSI on the area of the blotter affected by the press. Maintain this pressure for exactly one minute. Release the pressure on the press, open the press and remove the handsheet.

15 C) Handsheet Drying:

Place the handsheet on the polished surface of the sheet dryer (Valley Steam hot plate). Carefully lower the canvas cover over the sheet and fasten the 13 lb. dead weight to the lead filled brass tube. Allow the sheet to dry for 2 minutes. The surface temperature, with cover removed, should average 100.5 plus or minus 1 degree C. Remove the sheet from
20 the dryer and trim to the 7-1/2 inch X 7-1/2 inch. Weigh the sheet immediately.

Testing of Handsheets

Handsheets shall all be tested at the standard 50% humidity and 73 degree F temperature basis.

25 Bulk, Burst Index. Tear index and tensile index of the handsheets are tested according to TAPPI (Technical Association of Pulp and Paper Industry) test method (T220 om-88).

Brightness and color are tested with Technibrite Micro TB-1C (manufactured by
30 Technidyne Corporation, New Albany, Indiana, USA) following its Instruction Manual.

Bulk and Dry Density of an Absorbent Structure

From a handsheet prepared according to the procedure described herein, a strip of sample handsheet material, having a width of about 2 inches and a length of about 15 inches, was obtained by using a textile saw available, for example from Eastman, Machine Corp., Buffalo, New York. The sample strip was cut at least about 1 inch away from the edge of the handsheet so as to avoid edge effects. The sample strip was marked in about 10 millimeter intervals using water-soluble ink. To measure the bulk of the sample strip, a bulk meter accurate to at least about 0.01 millimeter, such as a bulk meter available from Mitutoyo Corporation, was used. An about one inch diameter platen was used to measure the bulk, with the platen being parallel to the base of the bulk meter. The bulk of the sample strip was measured in about 50 millimeter intervals along the length of the sample strip and then averaged. The average bulk of the sample strip was then used to calculate the dry density of the sample strip, using the weight and dimensions of the sample strip.

The basic raw material consists of market de-inked recycled fibers. These fibers are treated at 200 °F for 1, 2 and 4 minutes. After this steam treatment, the pulp was blown into a container by instantaneously releasing the steam pressure. The pulp obtained was made into handsheets for testing. Table 1 shows that this results in a product which exhibits both increased bulk and porosity and reduced tensile. Unfortunately, brightness is also reduced. This can be corrected by adding bleach materials to the recycle fibers prior to the steam explosion process. This is seen in the results given in Table 2.

Tables 3 and 4 show the increase of handsheet tensile strength with the addition of sodium hydroxide and sodium monochloroacetate to the fiber slurry prior to the steam explosion.

Table 1

Whole de-inked fibers: 80% consistency and dried at 80 °C.

Steam Explosion Conditions: 50% consistency at 200 °C for 2 Minutes.

Steam Explosion / Bleaching	H ₂ O ₂	0	0.5%	1%	2%	3%
	NaOH	0	0.2%	0.4%	0.6%	0.8%
PFI REVOLUTIONS		0	0	0	0	0
SI CONVERTED						
AVERAGE TEST DATA						
Specific Volume (cm ³ /g)		2.47	2.38	2.39	2.37	2.39
Tensile Index (Nm/g)		22.72	28.33	23.94	22.79	23.83
Tensile Energy Absorp. (J/m ²)		28.41	37.76	25.19	26.50	27.27
AVERAGE PHYSICAL TEST DATA						
C.S. Freeness (ml)		410	390	405	415	390
Bulk (in)		0.0058	0.0056	0.0056	0.0056	0.0056
Tensile (lbs)		7.72	9.63	8.14	7.75	8.10
Stretch (%)		3.037	3.243	2.622	2.845	2.785
Tensile Energy Absorb (ftlb/ft ²)		1.946	2.586	1.725	1.815	1.868
Porosity (Frazier) (cfm/ft ²)		53.0	41.0	48.6	49.6	47.8
AVERAGE OPTICAL TEST DATA						
(ISO) Brightness (%)		72.70	80.35	80.75	80.06	80.47
(ISO) Opacity (%)		84.61	81.25	80.46	80.99	80.79
Scattering Coefficient (m ² /kg)		41.84	41.23	40.45	41.05	40.96
Absorption Coefficient (m ² /kg)		0.92	0.49	0.44	0.47	0.45
L (%)		92.19	94.20	94.43	94.30	94.40
a (%)		-0.08	-0.63	-0.50	-0.40	-0.38
b (%)		6.97	4.29	4.37	4.67	4.51

Table 2

Whole de-inked fibers: 75% consistency and dried at 80 °C.

Steam Explosion Conditions	Control	200 °C	200 °C	200 °C
Pressure Vessel Time	-	1 Min	2 Min	4 Min
Bleaching Conditions	H ₂ O ₂	-	0.5%	1%
(10% consistency/60 °C/120 Min)	NaOH	-	0.4%	0.8%
PFI REVOLUTIONS	0	0	0	0
SI CONVERTED AVERAGE TEST DATA				
Specific Volume (cm ³ /g)	2.23	2.55	2.54	2.49
Tensile Index (Nm/g)	32.01	20.15	21.87	20.67
Tensile Energy Absorp. (J/m ²)	44.28	23.77	24.08	20.91
AVERAGE PHYSICAL TEST DATA				
C.S. Freeness (ml)	385	470	480	470
Bulk (in)	0.0053	0.0060	0.0060	0.0059
Tensile (lbs)	10.88	6.85	7.43	7.03
Stretch (%)	3.376	2.884	2.711	2.527
Tensile Energy Absorb (ftlb/ft ²)	3.033	1.628	1.649	1.432
Porosity (Frazier) (cfm/ft ²)	35.8	60.7	62.3	68.8
AVERAGE OPTICAL TEST DATA				
(ISO) Brightness (%)	81.93	79.06	79.67	80.42
(ISO) Opacity (%)	78.73	81.81	81.39	80.97
Scattering Coefficient (m ² /kg)	38.40	42.99	42.76	42.43
Absorption Coefficient (m ² /kg)	0.37	0.47	0.44	0.41
L (%)	94.78	94.41	94.61	94.76
a (%)	-0.98	-0.68	-0.74	-0.73
b (%)	4.16	5.71	5.55	5.23
pH				
Initial	-	11.3	11.6	11.4
Bleached Pulp Filtrate	-	10.6	11.0	10.9

Table 3**Reaction Mixture Used for Chemi-Steam Explosion Treatment**

Code #	LL-19 fiber (parts)	NaOH (parts)	ClCH₂COONa (parts)
Control	100	0	0
4189-1	100	2.2	8.6
4189-2	100	3	8.6
4189-3	100	4.4	17.2
4189-4	100	5.9	17.2
4189-5	100	6.7	25.8
4189-6	100	8.9	25.8

Table 4**Physical Properties of Handsheets****Made from Carboxymethylation-Steam Explosion Fibers**

Code #	Bulk (cc/g)	Dry Strength (g/3Inch)	Wet Strength (g/3Inch)	Wet Tensile/Dry Tensile (%)
Control	2.25	4754	1179	24.8
4189-1	2.84	4716	1396	29.6
4189-2	2.84	4488	1431	31.9
4189-3	2.88	4772	1422	29.8
4189-4	2.84	4732	1410	31.2
4189-5	2.8	4870	1534	31.5
4189-6	2.8	5028	1604	31.9

Basis weight of handsheet is 60 g/m² with addition of 1% Kymene

The above specification, examples and data provide a complete description of the manufacture and use of the composition of the invention. Since many embodiments of the invention can be made without departing from the spirit and scope of the invention, the invention resides in the claims hereinafter appended.

WE CLAIM:

1. A process for the production of a fiber having a permanently modified fiber morphology comprising a curl index of at least 0.2 resulting in a fiber with increased bulk, increased surface area and low density, the process comprising:
 - (a) subjecting the fiber to treatment with steam at super atmospheric temperature and pressure for a sufficient period of time to render the fiber morphology permanent; and
 - (b) explosively releasing the super atmospheric steam pressure.
2. The process of claim 1 wherein the fiber comprises a purified papermaking wood fiber.
3. The process of claim 1 wherein the fiber comprises a recycled papermaking wood fiber.
4. The process of claim 1 wherein the super atmospheric pressure and temperature comprises a temperature of about 130 to 250°C and a pressure of about 40 to 405 psi within a sealed continuous process container.
5. The process of claim 1 wherein the fiber with a permanent fiber morphology is blended with a purified papermaking wood fiber at a ratio of about 1 part of the papermaking wood fiber per each 0.01 to 100 parts of the fiber with the permanent fiber morphology.
6. An improved fiber material having increased bulk, increased surface area and low density comprising a blend of a fiber having a permanent fiber morphology comprising a curl index of at least 0.2, said fiber being the product of the process of first mixing a papermaking wood fiber with a fiber modifying compound for a predetermined period of time and subjecting the fiber to steam at super atmospheric temperature and pressure to obtain the permanent fiber morphology; with a refined papermaking wood fiber wherein there are about 0.01 to about 100 parts of the fiber having a permanent fiber morphology per each one part by weight of the refined papermaking fiber.

7. A tissue material formed from the fiber source of claim 6.